Revent by #

MEMORANDUM

TO:

Mr. Addison Rice

Anderson, Mulholland and Associates

DATE: February 21, 2015

FROM: R. Infante

FILE: 1502113

RE:

Data Validation

Air samples

SDG: 1502113A/1502113B

SUMMARY

Full validation was performed on the data for several gas samples analyzed for selected volatile organic compounds by method Compendium Method TO-15: Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999 and methane by ASTM method D-1946-modified. The samples were collected at the Bristol Myer Squib-Building 6 VI facility, Humacao, PR site on February 04 and 06, 2015 and submitted to Eurofins Air Toxics, Inc. of Folson, California that analyzed and reported the results under delivery groups (SDG) 1502113A/1502113B.

The sample results were assessed according to USEPA data validation guidance documents in the following order of precedence: Compendium Method TO-15. Determination Of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters And Analyzed By Gas Chromatography/Mass Spectrometry (GC/MS), January, 1999; Validating Air Samples. Volatile Organic Analysis of Ambient Air in Canisters by Method TO-15, (SOP # HW-31. Revision #4. October, 2006; and the QC criteria of the ASTM method D-1946-modified. The QC criteria and data validation actions listed on the data review worksheets are from the primary guidance document, unless otherwise noted.

In general the data is valid as reported and may be used for decision making purposes. The data results are acceptable for use. Results for Benzene in samples 1502113A-02A and 1502113A-03A were qualified as estimated (J) due to RPD for field duplicates outside method performance criteria of $\pm 25\%$.

SAMPLES

The samples included in the review are listed below

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B6-5SS	1502113A-01A	02/03/2015	Air	VOCs
B6-3SS	1502113A-02A	02/03/2015	Air	VOCs
B6-3SSD	1502113A-03A	02/03/2015	Air	VOCs
B6-4SS	1502113A-04A	02/03/2015	Air	VOCs

Client Sample ID	Lab. Sample ID	Collected Date	Matrix	Analysis
B6-5SS	1502113A-01A	02/03/2015	Air	Methane
B6-3SS	1502113A-02A	02/03/2015	Air	Methane
B6-3SSD	1502113A-03A	02/03/2015	Air	Methane
B6-4SS	1502113A-04A	02/03/2015	Air	Methane

REVIEW ELEMENTS

Sample data were reviewed for the following parameters, where applicable to the method

- o Agreement of analysis conducted with chain of custody (COC) form
- Holding time and sample preservation
- Gas chromatography/mass spectrometry (GC/MS) tunes
- Initial and continuing calibrations 0
- Method blanks/trip blanks/field blank 0
- Canister cleaning certification criteria 0
- Surrogate spike recovery 0
- Internal standard performance and retention times 0
- Field duplicate results 0
- Laboratory control sample/laboratory control sample duplicate (LCS/LCSD) results
- Quantitation limits and sample results

DISCUSSION

Agreement of Analysis Conducted with COC Request

Sample reports corresponded to the analytical request designated on the chain-of-custody form.

Holding Times and Sample Preservation

Sample preservation was acceptable.

Samples analyzed within method recommended holding time.

GC/MS Tunes

The frequency and abundance of bromofluorobenzene (BFB) tunes were within the QC acceptance criteria. All samples were analyzed within the tuning criteria associated with the method.

Initial and Continuing Calibrations

VOCs (Method TO-15)

The percent relative standard deviations (%RSDs) and response factors (RFs) of all target analytes were within the QC acceptance criteria in the initial calibration. Correlation coefficients (r2) of target analytes were within the QC acceptance criteria. Ongoing accuracy of the instrument was determined by the analysis of a continuing calibration standard.

Methane by ASTM method D-1946 (modified)

Initial and continuing calibrations meet method specific requirements. Initial calibration retention times meet method specific requirements.

Method Blank/Trip Blank/Field Blank

Target analytes were not detected in laboratory method blanks.

Summa canister met cleaning certification criteria.

No trip/field blank analyzed with this data package.

Surrogate Spike Recovery

The surrogate recoveries were within the laboratory QC acceptance limits in all samples analyzed.

Internal Standard Performance

VOCs and Methanol (TO-15)

Samples were spiked with the method specified internal standard. Internal standard are performance and retention times met the QC acceptance criteria in all sample analyses and calibration standards.

Laboratory/Field Duplicate Results

Field/laboratory duplicates were analyzed as part of this data set. Target analytes meet the RPD performance criteria of + 25 % for analytes 5 x SQL except for the followings:

Results for benzene in samples 1502113A-02A and 1502113A-03A were qualified as estimated (j) due to RPD for field duplicates outside method performance criteria of \pm 25 %.

LCS/LCSD Results

VOCs and Methane

LCS/LCSD (blank spike) were analyzed by the laboratory associated with this data package. Recoveries and RPD within laboratory control limits.

Quantitation Limits and Sample Results

Dilutions were performed on TO-15 samples (see worksheet).

Calculations were spot checked.

Certification

The following samples 1502113A-01A; 1502113A-02A; 1502113A-03A; 1502113A-04A; 1502113B-01A; 1502113B-02A; 1502113B-03A; and 1502113B-04A were analyzed following standard procedures accepted by regulatory agencies. The quality control requirements met the methods criteria except in the occasions described in this document. The results are valid. Some of the results were qualified.

Rafael Infante

Chemist License 1888



Client Sample ID: B6-5SS Lab ID#: 1502113A-01A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name: Dil. Factor:	v021017 24.2	Date of Collection: 2/4/15 3:49:00 PM Date of Analysis: 2/10/15 07:20 PM			
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit (ug/m3)	Amount (ug/m3)	
	2.4	1.9 J	7.7	6.1 J	
Benzene	2.4	14	10	59	
Ethyl Benzene	2.4	39	- 10	170	
m,p-Xylene	2.4	11	10	49	
o-Xylene	2.4	880	9.1	3300	
Toluene		390	30	960	
2-Propanol	12			150	
4-Methyl-2-pentanone	2.4	36	9.9		
Acetone	12	150	29	360	

J = Estimated value.

Container Type: 1 Liter Summa Camste	Method	
Surrogates	%Recovery	Limits
	96	70-130
1,2-Dichloroethane-d4	99	70-130
Toluene-d8	86	70-130
4-Bromofluorobenzene	95	





Client Sample ID: B6-3SS Lab ID#: 1502113A-02A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name: Dil. Factor:	v021016 2.29	Date of Collection: 2/4/15 4:37:00 PM Date of Analysis: 2/10/15 06:45 PM			
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit A (ug/m3) (
	0.23	4.0 🕽	0.73	13	
Benzene	0.23	12	0.99	54	
Ethyl Benzene	0.23	1.4	0.99	5.9	
m,p-Xylene	0.23	0.42	0.99	1.8	
o-Xylene		6.7	0.86	25	
Toluene	0.23			140	
2-Propanol	1.1	58	2.8		
4-Methyl-2-pentanone	0.23	0.65	0.94	2.7	
Acetone	1.1	74	2.7	180	

Container Type: 1 Liter Summa Camster	Method	
Surrogates	%Recovery	Limits
	102	70-130
1,2-Dichloroethane-d4	98	70-130
Toluene-d8 4-Bromofluorobenzene	90	70-130





Client Sample ID: B6-3SSD Lab ID#: 1502113A-03A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name: Dil. Factor:	v021018 2,29	Date of Collection: 2/4/15 4:37:00 PM Date of Analysis: 2/10/15 08:21 PM			
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit Am (ug/m3) (ug		
	0.23	1.2 3	0.73	3.7	
Benzene	0.23	13	0.99	57	
Ethyi Benzene	0.23	0.98	0.99	4.2	
m,p-Xylene		0.43	0.99	1.9	
o-Xylene	0.23	7.0	0.86	26	
Toluene	0.23		2.8	150	
2-Propanol	1.1	61		2.8	
4-Methyl-2-pentanone	0.23	0.68	0.94		
Acetone	1.1	76	2.7	180	

Container Type: 1 Litter Summa Caniste	Method	
Surrogates	%Recovery	Limits
1,2-Dichloroethane-d4	104	70-130
Toluene-d8	97	70-130
4-Bromofluorobenzene	90	70-130





Client Sample ID: B6-4SS Lab ID#: 1502113A-04A

MODIFIED EPA METHOD TO-15 GC/MS FULL SCAN

File Name: Dil. Factor:	v02101 9 12.4	Date of Collection: 2/6/15 2:22:00 PM Date of Analysis: 2/10/15 08:55 PM			
Compound	Rpt. Limit (ppbv)	Amount (ppbv)	Rpt. Limit Amou (ug/m3) (ug/m		
Compound	1.2	Not Detected	4.0	Not Detected	
Benzene		1.8	5.4	8.0	
Ethyl Benzene	1.2		5.4	48	
m,p-Xylene	1.2	11		18	
o-Xylene	1.2	4.1	5.4		
Toluene	1.2	2.5	4.7	9.5	
	6.2	1900 E	15	4700 E	
2-Propanol		0.43 J	5.1	1.8 J	
4-Methyl-2-pentanone	1.2		15	400	
Acetone	6.2	170	15	400	

E = Exceeds instrument calibration range.

Container Type: 1 Liter Summa Canister (100% Certified)

Surrogates	%Recovery	Limits
1,2-Dichloroethane-d4	97	70-130
Toluene-d8	97	70-130
4-Bromofluorobenzene	88	70-130



Method

J = Estimated value.

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Client Sample ID: B6-5SS Lab ID#: 1502113B-01A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021022 2.42	Date of Collect Date of Analy	tion: 2/4/15 3:49:00 PM sis: 2/10/15 07:57 PM
Dil. Factor:		Rpt. Limit (%)	Amount (%)
Compound			0.00038
Methane		0.00024	0.0000





Client Sample ID: B6-3SS Lab ID#: 1502113B-02A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9021021 2.29	Date of Collection: 2/4/15 4:37:00 PM Date of Analysis: 2/10/15 07:14 PM		
		Rpt. Limit (%)	Amount (%)	
Compound Methane		0.00023	0.00019 J	

J = Estimated value.





Client Sample ID: B6-3SSD Lab ID#: 1502113B-03A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name: Dil. Factor:	9021023 2,29	Date of Collection: 2/4/15 4:37:00 PM Date of Analysis: 2/10/15 08:34 PM		
		Rpt. Limit (%)	Amount (%)	
Compound Methane		0.00023	0.00022 J	

J = Estimated value.





Client Sample ID: B6-4SS Lab ID#: 1502113B-04A

NATURAL GAS ANALYSIS BY MODIFIED ASTM D-1946

File Name:	9021024 2.47	Date of Collect Date of Analy	Date of Collection: 2/6/15 2:22:00 PM Date of Analysis: 2/10/15 09:06 PM		
		Rpt. Limit (%)	Amount (%)		
Compound Methane		0.00025	0.00021 J		

J = Estimated value.





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01A B6-3550		2/4/15	1637		30	5		
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	Project Number:1502113A
	Date:02/04/15_&_02/06/2015
REVIEW OF VOLATILE ORGA The following guidelines for evaluating volatile organics we actions. This document will assist the reviewer in using prodecision and in better serving the needs of the data users. The USEPA data validation guidance documents in the followin "Compendium Method TO-15. Determination of Volatile Orga Specially-Prepared Canisters and Analyzed By Gas Chro January, 1999"; USEPA Hazardous Waste Support Branch Analysis of Ambient Air in Canisters by Method TO-15, (SOP a QC criteria and data validation actions listed on the data review document, unless otherwise noted. The hardcopied (laboratory name) _EurofinsAir_Toxicsreviewed and the quality control and performance data summaria.	ere created to delineate required validation of the sessional judgment to make more informed to example results were assessed according to any order of precedence: QC criteria from anic Compounds (VOCs) In Air Collected International Collected In
Lab. Project/SDG No.:1502113A No. of Samples:4_	Sample matrix:Air
Trip blank No.: Field blank No.: Equipment blank No.: Field duplicate No.:B6-3SS/B6-3SSD X Data CompletenessX Holding TimesX GC/MS TuningX Internal Standard PerformanceX Blanks	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Definition of Qualifiers: J- Estimated results U- Compound not detected R- Rejected data UJ- Estimated nondetect Reviewer: Au Date: 02/21/2015	

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
<u></u>		
	<u> </u>	

All criteria were metX
Criteria were not met
and/or see below

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pН	ACTION
<u> </u>			_	
				t alding time
	All samples analyzed w	vithin the recommended	method	noiding time

<u>Criteria</u>

Aqueous samples - 14 days from sample collection for preserved samples (pH ≤ 2, 4°C), no air bubbles.

Aqueous samples - 7 days from sample collection for unpreserved samples, 4°C, no air bubbles.

Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 ± 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

All criteria were metX
Criteria were not met see below

GC/MS TUNING

If no, use professional judgment to determine whether the associated data should be accept	ed.				
XBFB tuning was performed for every 24 hours of sample analysis.					
XThe BFB performance results were reviewed and found to be within the specified criteria.					
The assessment of the tuning results is to determine if the sample instrumentation is within t standard tuning QC limits	the				

If mass calibration is in error, all associated data are rejected.

All criteria were metX
Criteria were not met
and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:	01/23/15
Dates of continuing calibrat	
Instrument ID numbers:	
	Air/low
Matrix/Level:	

DATE	LAB ID#	FILE	CRITERIA OUT RFs, %RSD, %D, r	COMPOUND	SAMPLES AFFECTED
Initial and	continui	ng calib	rations meet method s requirements.	pecific requirements. Initia	al calibration retention
unes nee	LINGUIOU	Specialo	Tequilottions.		
	_				

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC.

All %RSD must be ≤ 15 % regardless of method requirements for CCC.

All %Ds must be $\leq 30\%$ regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of ≥ 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ). If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE Analyzed	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
Ail_method	i_blank_meeth_	 _method_specif	ic_criteria	
Field <u>/</u> Equipmen				
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
No_field/trip/eq	uipment_blanks	_analyzed_with	n_this_data_package	

All criteria were metX
Criteria were not met
and/or see below

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is ≥ SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
SOUNCEPEEVEE					3.53
			- C		
		-15			
		1			
				1	

All criteria were met _X
Criteria were not met
and/or see below

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery.

Matrix: solid/aqueous

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Ω2	. 24	Pl	-		п

SURROGATE COMPOUND

ACTION

1,2-DICHLOROETHANE- Toluene- 4-BFB d8

_Surrogate_recoveries_within_laboratory	_control_limits
	,
QC Limits* (Air)	
LL to UL70_to_130	_70to_13070to_130

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

QUALITY	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J	J	J
Nondetects results	R	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

	PD of the compounds		not meet the criteria. Matrix/Level:			
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION	
	_are_not_required_as	_part_of_l	Method_	TO-15;_blank_sp	ike_used_to_assess	

- * QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- * If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

All criteria were met _____ Criteria were not met and/or see below __N/A__

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Le	vel/Unit	
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

All criteria were metX	
Criteria were not met	
and/or see below	

QC LIMIT

LABORATORY CONTROL SAMPLE (LCS) ANALYSIS VIII.

This data is generated to determine accuracy of the analytical method for various matrices.

LCS Recoveries Criteria 1.

Where LCS spiked with the same analyte at the same concentrations as the MS/MSD? Yes or No. If no make note in data review memo. List the %R of compounds which do not meet the criteria

	LCS ID	COMPOUND	% R	QC LIMIT
LCS/LCSI laboratory)_(Blank_spike) _control_limits	_analyzed_in_this_data_	package,_recoveries_an	d_RPD_within

- QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper
- If QC limits are not available, use limits of 70 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

Frequency Criteria: 2.

Where LCS analyzed at the required frequency and for each matrix? Yes or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		All criteria were met Criteria were not met and/or see belowX
IX.	LABORATORY DUPLICATE PRECISION	
	Sample IDs: _1502113A-02A/1502113A-03A	Matrix:Air

Field duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information.

Suggested criteria: RPD ± 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
Benzene	0.23	4.0	1.2	108 %	Qualify results in sample and duplicate
RPE) within the m	ethod perfor	 mance criteria f	or all other	r analytes.

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metX
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
_Internal_s _and_calib	tandard_area_and_re	etention_times_	within_laboratory	_control_limits_for_	both_samples
Actions:					

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results		ACCEPT
Nondetected results	R	ACCEPT

If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were metX
Criteria were not met
and/or see below

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

15022113-01A

Acetone

RF = 1.15451

[] = (271643)(5.0)/(190063)(1.15451)

= 6.19 ppbv OK

All criteria were metX	
Criteria were not met	
and/or see below	

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
Dilution was pe	erformed on samples B	6-5SS (24.2 x) and B6-4SS (12.4) due to the presence of
high level targe	t species.	
-		

B.	Percent	Solids
----	---------	--------

List samples wh	ich have ≤ 50 % solids	1	

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ) If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects

(R)

	Project Number:1502113B
	Date:02/04/2015_&_02/06/2015
REVIEW OF VOLATILE ORGONE The following guidelines for evaluating volatile organics was actions. This document will assist the reviewer in using plecision and in better serving the needs of the data users. The JSEPA data validation guidance documents in the following 0-1946 method for measuring permanent gases and light samples using gas chromatography (GC) and a thermal conclustration (FID). Validating Air Samples. Volatile Organic Air O-15, (SOP # HW-31. Revision #4. October, 2006). The Grant data review worksheets are from the primary guidance do the hardcopied (laboratory name) _Eurofinseviewed and the quality control and performance data summer process.	were created to delineate required validation rofessional judgment to make more informed the sample results were assessed according to order of precedence: QC criteria from ASTM thydrocarbons in refinery and other source ductivity detector (TCD) and/or flame ionization nalysis of Ambient Air in Canisters by Method QC criteria and data validation actions listed or occument, unless otherwise noted. data package received has been
.ab. Project/SDG No.:1502113B	Sample matrix:Air
No. of Samples:4	
rip blank No.: Field blank No.: Equipment blank No.: Field duplicate No.: B63SS/B63SSD X Data CompletenessX Holding TimesN/A_ GC/MS TuningN/A_ Internal Standard PerformanceX BlanksN/A_ Surrogate RecoveriesN/A_ Matrix Spike/Matrix Spike Duplicate Diverall Comments:Methane_by_ASTM_method_D-194	X Laboratory Control SpikesX Field DuplicatesX CalibrationsX Compound IdentificationsX Compound QuantitationX Quantitation Limits
Definition of Qualifiers: - Estimated results J- Compound not detected R- Rejected data JJ- Estimated nondetect	
Reviewer: <u>Afail Mauf</u> Date:02/21/2015	

DATA COMPLETENESS

MISSING INFORMATION	DATE LAB. CONTACTED	DATE RECEIVED
	AND THE STATE OF T	
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•	# ·	

All criteria were metX	
Criteria were not met	
and/or see below	

HOLDING TIMES

The objective of this parameter is to ascertain the validity of the results based on the holding time of the sample from time of collection to the time of analysis.

Complete table for all samples and note the analysis and/or preservation not within criteria

SAMPLE ID	DATE SAMPLED	DATE ANALYZED	pН	ACTION
A	li samples analyzed w	rithin the recommender	d method	nolding time
*				

Criteria

Aqueous samples – 14 days from sample collection for preserved samples (pH \leq 2, 4°C), no air bubbles.

Aqueous samples – 7 days from sample collection for unpreserved samples, 4°C, no air bubbles. Soil samples- 7 days from sample collection.

Cooler temperature (Criteria: 4 + 2 °C): N/A – summa canisters

Actions

If the VOCs vial(s) have air bubbles, estimate positive results (J) and reject nondetects (R).

If the % solids of soil samples is 10-50%, estimates positive results (J) and nondetects (UJ)

If the % solid of soil samples is < 10%, estimate positive results (J) and reject nondetects (R).

If holding times are exceeded but < 14 days beyond criteria, estimate positive results (J) and nondetects (UJ).

If holding times are exceeded but < 28 days beyond criteria, estimate positive results (J) and reject nondetects (R).

If holding times are grossly exceeded (> 28 days beyond criteria), reject all results (R).

If samples were not iced or if the ice were melted (> 10°C), estimate positive results (J) and nondetects (UJ).

		Crit	All criteria were metN/A eria were not met see below
GC/MS TUNING			
The assessment standard tuning C		determine if the sample instru	mentation is within the
N/A_ The BFB	performance results were	reviewed and found to be within	the specified criteria.
N/A_ BFB tunir	ng was performed for every	24 hours of sample analysis.	
If no, use profest qualified or reject	• •	ine whether the associated dat	ta should be accepted,
List	the	samples	affected:
If mass calibration	n is in error, all associated (data are rejected.	

Note: Samples analyzed using GC with either TCD or FID detection.

4

All criteria were metX
Criteria were not met
and/or see below

CALIBRATION VERIFICATION

Compliance requirements for satisfactory instrument calibration are established to ensure that the instrument is capable of producing and maintaining acceptable quantitative data.

Date of initial calibration:_	05/08/14
Dates of continuing calibration	ation:_02/10/15
Instrument ID numbers:	GC-9
Matrix/Level:	Air/low

DATE	LAB	FILE	CRITERIA OUT	COMPOUND	SAMPLES
	ID#		RFs, %RSD, %D, r		AFFECTED
Initial and o	ontinuir	ng calibi	ations meet method sp	ecific requirements. Initial	calibration retention
	times meet method specific requirements.				

Criteria

All RFs must be > 0.05 regardless of method requirements for SPCC. All %RSD must be ≤ 15 % regardless of method requirements for CCC.

All %Ds must be < 30% regardless of method requirements for CCC.

Method TO-15 does not specify criterion for the curve correlation coefficient (r). A limit for r of > 0.995 has therefore been utilized as professional judgment.

Actions

If any compound has an initial RF or a continuing RF of < 0.05, estimate positive results (J) and reject nondetects (R), regardless of method requirements.

If any compound has a %RSD > 15%, estimate positive results (J) and use professional judgment to qualify nondetects.

If any compound has a %RSD > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and reject nondetects (R).

If any compound has a % D > 30%, estimate positive results (J) and nondetects (UJ).

If any compound has a % D > 90%, estimate positive results (J) and reject nondetects (R).

If any compound has r > 0.995, estimate positive results and nondetects.

A separate worksheet should be filled for each initial curve

All criteria were metX
Criteria were not met
and/or see below

V A. BLANK ANALYSIS RESULTS (Sections 1 & 2)

The assessment of the blank analysis results is to determine the existence and magnitude of contamination problems. The criteria for evaluation of blanks apply only to blanks associated with the samples, including trip, equipment, and laboratory blanks. If problems with any blanks exist, all data associated with the case must be carefully evaluated to determine whether or not there is an inherent variability in the data for the case, or if the problem is an isolated occurrence not affecting other data.

List the contamination in the blanks below. High and low levels blanks must be treated separately.

Laboratory blanks

DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
All_method	 d_blank_meeth	 _method_speci		
Summa_ca			ation_criteria	
Field/Equipmen				
DATE ANALYZED	LAB ID	LEVEL/ MATRIX	COMPOUND	CONCENTRATION UNITS
No_field/trip/equ	uipment_blanks	_analyzed_with	_this_data_package	
	b			

		•		

All criteria were met _X_	
Criteria were not met	
and/or see below	

VB. BLANK ANALYSIS RESULTS (Section 3)

Blank Actions

Action Levels (ALs) should be based upon the highest concentration of contaminant determined in any blank. Do not qualify any blank with another blank. The ALs for samples which have been diluted should be corrected for the sample dilution factor and/or % moisture, where applicable. No positive sample results should be reported unless the concentration of the compound in the samples exceeds the ALs:

ALs = 10x the amount of common contaminants (methylene chloride, acetone, 2-butanone, and toluene)

ALs = 5x for any other compounds

Specific actions are as follows:

If the concentration is < sample quantitation limit (SQL) and \le AL, report the compound as not detected (U) at the SQL.

If the concentration is \geq SQL but \leq AL, report the compound as not detected (U) at the reported concentration.

If the concentration is \geq SQL and > AL, report the concentration unqualified.

Notes:

High and low level blanks must be treated separately

Compounds qualified "U" for blank contamination are still considered "hits" when qualifying for calibration criteria.

CONTAMINATION SOURCE/LEVEL	COMPOUND	CONC/UNITS	AL/UNITS	SQL	AFFECTED SAMPLES
					The state of the s
				7	
	The same of the sa				
545					
Special Control					

All criteria were met _	N/A
Criteria were not met	
and/or see below	

SURROGATE SPIKE RECOVERIES

Laboratory performance of individual samples is established by evaluation of surrogate spike recoveries. All samples are spiked with surrogate compounds prior to sample analysis. The accuracy of the analysis is measured by the surrogate percent recovery. Since the effects of the sample matrix are frequently outside the control of the laboratory and may present relatively unique problems, the validation of data is frequently subjective and demands analytical experience and professional judgment.

List the percent recoveries (%Rs) which do not meet the criteria for surrogate recovery. Matrix: solid/aqueous

SAMPLE ID	SURROGATE COMPOUND	ACTION
_Surrogate_standards_not	_required_by_the_method	
QC Limits* (Air) LL_to_ULto_	to to	

- QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.
- If QC limits are not available, use limits of 80 120 % for aqueous and 70 130 % for solid samples.

Actions:

SAMPLE ID

QUALITY	0/D - 400/		
	%R < 10%	%R = 10% - LL	%R > UL
Positive results	J		133.2
Nondetects results	D		J
Trondottota resulta	I K	UJ	Accept

Surrogate action should be applied:

If one or more surrogate in the VOC fraction is out of specification, but has a recovery of > 10%.

If any one surrogate in a fraction shows < 10 % recovery.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. A MATRIX SPIKE/MATRIX SPIKE DUPLICATE (MS/MSD)

This data is generated to determine long term precision and accuracy in the analytical method for various matrices. This data alone cannot be used to evaluate the precision and accuracy of individual samples. If any % R in the MS or MSD falls outside the designated range, the reviewer should determine if there are matrix effects, i.e. LCS data are within the QC limits but MS/MSD data are outside QC limit.

1. MS/MSD Recoveries and Precision Criteria

The laboratory should use one MS and a duplicate analysis of an unspiked field sample if target analytes are expected in the sample. If target analytes are not expected, MS/MSD should be analyzed.

List the %Rs, RPD of the compounds which do not meet the criteria.

Sample ID:			Matrix/Level:			
MS OR MSD	COMPOUND	% R	RPD	QC LIMITS	ACTION	
_MS/MSD_are_r _accuracy	not_required_as_part	_of_ASTM	1-method	I_D-1946;_blank_	_spike_used_to_as	Sess_

If QC limits are not available, use limits of 70 - 130 %.

Actions:

QUALITY	%R < LL	%R > UL
Positive results	J	J
Nondetects results	R	Accept

MS/MSD criteria apply only to the unspiked sample, its dilutions, and the associated MS/MSD samples:

If the % R for the affected compounds were < LL (or 70 %), qualify positive results (J) and nondetects (UJ).

If the % R for the affected compounds were > UL (or 130 %), only qualify positive results (J).

If 25 % or more of all MS/MSD %R were < LL (or 70 %) or if two or more MS/MSD %Rs were < 10%, qualify all positive results (J) and reject nondetects (R).

A separate worksheet should be used for each MS/MSD pair.

QC limits are laboratory in-house performance criteria, LL = lower limit, UL = upper limit.

All criteria were met
Criteria were not met
and/or see belowN/A

VII. B MATRIX SPIKE/MATRIX SPIKE DUPLICATE

MS/MSD - Unspiked Compounds

It should be noted that Method TO-15 does not specify a MS/MSD criteria for the unspiked compounds in the sample. A %RSD of < 50% has therefore been utilized as professional judgment.

If all target analytes were spiked in the MS/MSD, this review element is not applicable.

List the %RSD of the compounds which do not meet the criteria.

Sample ID:		Matrix/Level/Unit_			
COMPOUND	SAMPLE CONC.	MS CONC.	MSD CONC.	% RSD	ACTION

Actions:

^{*} If the % RSD > 50, qualify the positive result in the unspiked samples as estimated (J).

^{*} If the % RSD is not calculated (NC) due to nondetected value, use professional judgment to qualify the data.

			C	l criteria were metX riteria were not met rd/or see below
VIII.	LABORATORY CONTROL S.	AMPLE (LCS) AN	ALYSIS	
	This data is generated to dete	ermine accuracy o	f the analytical metho	od for various matrices.
	1. LCS Recoveries Crite	eria		
	Where LCS spiked with the s Yes or No. If no make note in List the %R of compounds whi	uala review memi)	ions as the MS/MSD?
	LCS ID COM	POUND	% R	QC LIMIT
L(wi	CS/LCSD_(Blank_spike)_analyz ithin_laboratory_control_limits	ed_in_this_data_	oackage;_recoveries	_and_RPD
	QC limits are laboratory limit. If QC limits are not available.			ower limit, UL = upper
ctions:	and mot ara		170-130%.	
	QUALITY	%R < LL	%R > UL	
	Positive results	J	J	
	Nondetects results	R	Accept	

All analytes in the associated sample results are qualified for the following criteria.

If 25 % of the LCS recoveries were < LL (or 70 %), qualify all positive results (j) and reject nondetects (R).

If two or more LCS were below 10 %, qualify all positive results as (J) and reject nondetects (R).

2. Frequency Criteria:

Where LCS analyzed at the required frequency and for each matrix? <u>Yes</u> or No. If no, the data may be affected. Use professional judgment to determine the severity of the effect and qualify data accordingly. Discuss any actions below and list the samples affected.

		All criteria were metX Criteria were not met and/or see below
IX.	FIELD/LABORATORY DUPLICATE PRECISION	
	Sample ID_1502113B-02A/1502113B-03B	Matrix:Air

Field/laboratory duplicates samples may be taken and analyzed as an indication of overall precision. These analyses measure both field and lab precision; therefore, the results may have more variability than laboratory duplicates which only laboratory performance. It is also expected that soil duplicate results will have a greater variance than water matrices due to difficulties associated with collecting identical field duplicate samples.

The project QAPP should be reviewed for project-specific information. Suggested criteria: RPD \pm 25% for air samples. If both samples and duplicate are <5 SQL, the RPD criteria is doubled.

COMPOUND	SQL	SAMPLE CONC.	DUPLICATE CONC.	RPD	ACTION
RPD for field duplic	cates within la		trol limits. RPD atory control lim		ratory duplicate (LCS/LCSD)

Actions:

Qualify as estimated positive results (J) and nondetects (UJ) for the compound that exceeded the above criteria. For organics, only the sample and duplicate will be qualified.

If an RPD cannot be calculated because one or both of the sample results is not detected, the following actions apply:

If one sample result is not detected and the other is greater than 5x the SQL qualify (J/UJ).

If one sample value is not detected and the other is greater than 5x the SQL and the SQLs for the sample and duplicate are significantly different, use professional judgment to determine if qualification is appropriate.

If one sample value is not detected and the other is less than 5x, use professional judgment to determine if qualification is appropriate.

If both sample and duplicate results are not detected, no action is needed.

All criteria were metN/A
Criteria were not met
and/or see below

X. INTERNAL STANDARD PERFORMANCE

The assessment of the internal standard (IS) parameter is used to assist the data reviewer in determining the condition of the analytical instrumentation.

List the internal standard area of samples which do not meet the criteria.

- * Area of +40% or -40% of the IS area in the associated calibration standard.
- * Retention time (RT) within ± 0.06 seconds of the IS area in the associated calibration standard.

DATE	SAMPLE ID	IS OUT	IS AREA	ACCEPTABLE RANGE	ACTION
_Internal_st _method	andard_not_required	i_by_the_metho	odSamples_qua	ntified_by_externa	l_standard
Actions:	:				

1. IS actions should be applied to the compound quantitated with the out-of-control ISs

QUALITY	IS AREA < -40%	IS AREA > + 40%
Positive results	J	J
Nondetected results	R	ACCEPT

2. If a IS retention time varies more than 0.330 seconds, the chromatographic profile for that sample must be examined to determine if any false positive or negative exists. For shifts of a large magnitude, the reviewer may consider partial or total rejection of the data for the sample fraction.

All criteria were met __X__ Criteria were not met and/or see below ____

XII. SAMPLE QUANTITATION

The sample quantitation evaluation is to verify laboratory quantitation results. In the space below, please show a minimum of one sample calculation:

1502113B-01A

Methane

RF = 159808871

[] = [(25416)/(159808871)] * 2.42

= 3.84 X 10-4 % OK

All criteria were met	Χ_
Criteria were not met	
and/or see below	

XII. QUANTITATION LIMITS

A. Dilution performed

SAMPLE ID	DILUTION FACTOR	REASONS FOR DILUTION
All samples dilu	rted by a factor of less th	ian 2.5

B.	Percent Solids
----	----------------

List samples which have ≤ 50 % solids	

Actions:

If the % solids of a soil sample is 10-50%, estimate positive results (J) and nondetects (UJ) If the % solids of a soil sample is < 10%, estimate positive results (J) and reject nondetects (R)